

Gas Mixing Station User Manual

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July 2025

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1 Introduction

The Gas Mixing Station (GMS) can mix up to 4 different gases in a volume ratio to supply to our detectors. This is a versatile system that will enable us to fill our detectors with either one out of four gases, combinations of any of them, specific volumes, etc. This is a quick manual to summarize the important information from the original user's manual. Any questions not answered in this text can be answered using the original users manual, which should also be stored near the GMS.

The GMS is shown below in figure 1 and its individual parts are labeled. The Command Module (CM) is wired to GMS's four Mass Flow Controllers (MFCs). Each MFC is specifically calibrated for a different gas, which can be seen on its label. The specifics of each channel are shown in

table 1. These MFCs then connect to one central exit channel that is outfitted with a variable-pressure emergency relief valve and a pressure gauge before exiting through a helical-baffle filled 1/4 inch tube.

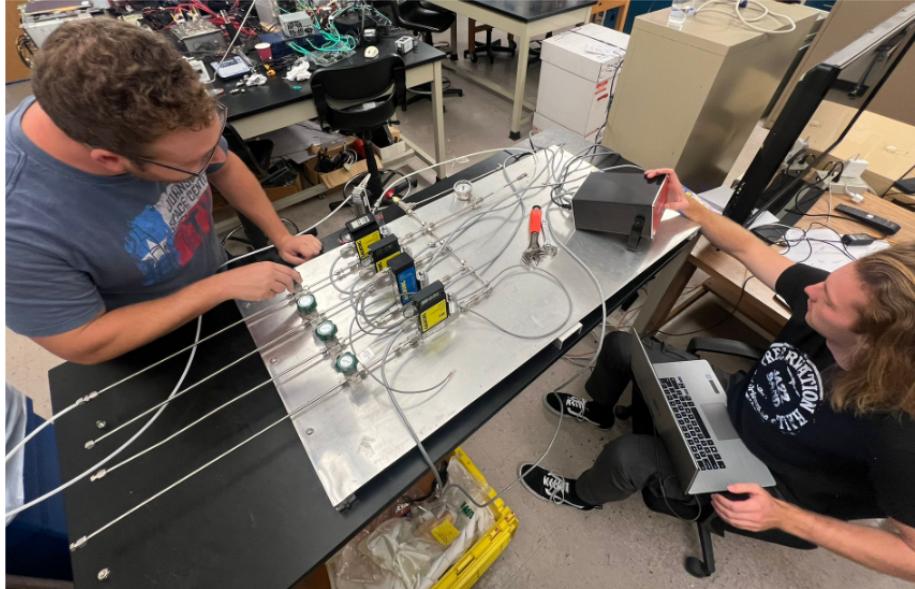


Figure 1: The GMS with its CM and four MFC channels

| Channel Number | Calibrated Gas | Max Flow Rate [ml/min] |
|----------------|----------------|------------------------|
| 1 | N ₂ | 200 |
| 2 | N ₂ | 50 |
| 3 | Ar | 50 |
| 4 | Xe | 10 |

Table 1: Description of each channel's calibrated gas and max flow rate

2 How to Operate the Command Module

The command module (CM) is used to control the four MFCs. The MFCs are sensitive devices and require at least 15 minutes of on time before being used. This warm-up period is crucial for accurate flow rates from the MFCs. Issues with fluctuations in the MFCs are discussed in chapter 4.2.

When the CM is turned on, the display will eventually show the current flow rate in units of % total flow rate of each channel as well as there reference mode, shown in Figure 2. Under the display there are four buttons that are used to navigate the CM: ESC, UP, DOWN, and ENTER. Each button changes functions depending on which screen is currently displayed.



Figure 2: Main CM display menu. Channels 1 and 2 are set to reference Ratio Mode (R), and channels 3 and 4 are set to internal reference (I).

From the starting screen, ESC is used to control all of the values at once. This can be useful if you need to close all of the channels at once or need to set all of the channels to their internally set value. UP is used to view the current status of all channels at once. By continuing to press UP one can cycle through various units and parameters for each channel.

DOWN and ENTER are used to set parameters and control the various channels independently. When either button is pressed, the first screen that will pop up will be to select which channel you would like to alter. DOWN allows the user to set parameters relating to the gas being measured, such as density, channel displayed units, and MFC threshold source. The density is only used to convert volume-based units to mass flow rates on the MFC display. It cannot be used to change the MFCs response to the gas being flowed through that channel. Changing the MFC threshold source allows the MFC to be controlled independently via an internal trigger set via the CM, an external trigger which would need to be set up via a wire directly to the MFC, or in ratio mode in tandem with the other N channels involved. The engineering units can be altered to whatever is convenient for the experiment. For the normal flow rates associated with detector work, Standard Cubic Centimeters per Minute (SCCM) is used. This is equivalent to measuring flow rate in milliliters per minute.

ENTER is used to control each channel's flow rate and status. The flow rate can be set anywhere between zero and the max flow rate in the units specified. Then, you can alter the status of the channel, choosing either open, closed, or auto. Auto refers to setting the channel to its internal threshold value set in the previous screen.

2.1 Controlling Channels

Each channel can be controlled independently using the menus offered with ENTER as long as the channels are not in ratio mode (explained in section 2.2). After selecting a channel, the flow rate can be set using + (UP) and - (DOWN). (*Note: Depending on the current units, one button press may not visually change the set flow rate, but it is changing the actual value, just not to a precision currently being displayed. To increase precision it is recommend to set units to SCCM.*) After the flow rate is set, the control mode can be changed by clicking NEXT (ESC) and then choosing either CLOSE (UP), OPEN (DOWN), or AUTO (ENTER). Auto is the only control mode which uses

the set flow rate. In order for auto mode to work, the channel reference must be set to Internal (I). Clicking ESC will show the totalizer (see section 2.3) status with the options to either reset the totalizer or exit to the starting screen.

2.2 Ratio Mode

Ratio mode allows the user to control two or more channels at once in tandem to set specific ratios of gases. First, each channel's reference mode must be set to program ratio. To do this from the starting screen click DOWN, select a channel, hit GENER. (ESC), then click ENTER until the top left reads PR RATIO. Once back on the starting screen, the flow rate of the selected channel should have an R next to it, indicating it is referencing ratio mode. Now ratio mode itself must be enabled.

Ratio mode can be toggled on or off by pressing DOWN and choosing any channel. Once the channel has been selected, press the button for program and then select ratio. From here, the ratio mode setting can be enabled or disabled. Once ratio mode is enabled for the selected channel, hitting NEXT (ESC) will allow the specific ratio to be set for that channel. The CM shows the total percent of all channels in the top right of the display. This value needs to be 100% for the machine to work properly. If all four channels are not being used, then the unused channels need to be set to 0% and disabled. Hitting NEXT (ESC) again cycles through the channels.

When ratio mode is enabled, only the flow rate in channel 1 can be manually changed. All other channels set to reference ratio mode will automatically adjust their flow rates relative to channel 1.

2.2.1 Examples with Ratio Mode and Calibration Curves

There are two main situations needing ratio mode that are commonly used in this lab. You may need a specific gas ratio for a detector, and we have the channels already calibrated for the gases, or you need to flow a different gas through the channel than it was calibrated for. Calibration curves will ensure the MFC flow the correct amount of gas with a different gas than initially calibrated for. This concept is used in other aspects as well, potentially with setting the ratio value itself.

If needing a gas ratio that can be made with the channels on the machine (N₂, Ar, or Xe), then the setup is fairly straightforward. For example, let's assume we want 40% N₂ and 60% Ar Gas. Using DOWN, toggle ratio mode on for channels 1 and 3 and set the ratios to the respective values. Remember to zero out the other two channels so the total is 100%. If using a ratio calibration curve, find the new input ratio value that corresponds to the ratio you want to output into the detector. Using the example graph below, we see that to obtain a 40:60 ratio for N₂:Ar, we want to set the machine to 41.4:58.6 for more accurate results.

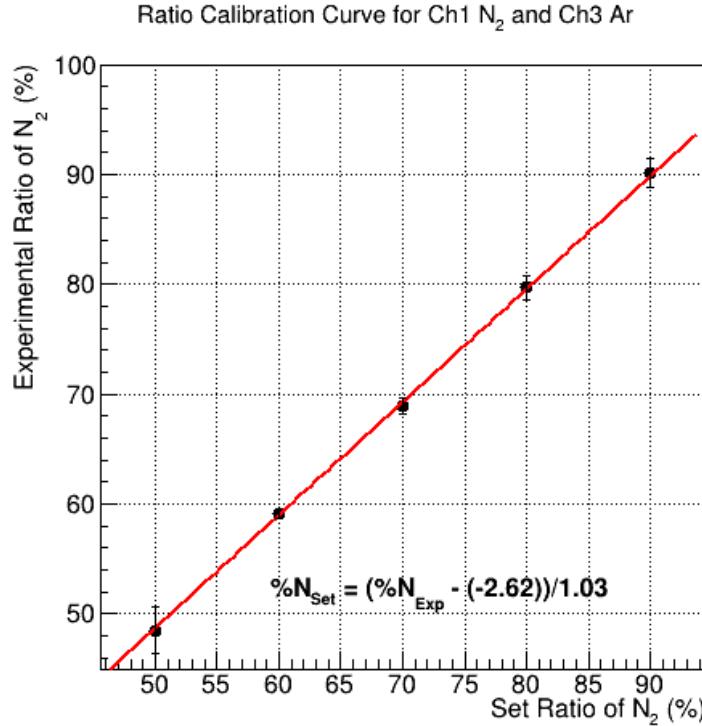


Figure 3: Ratio calibration curve for N₂:Ar. Please note that this is NOT a real calibration curve. This plot was made specifically as a visual aid for this example. Real calibration curves will be listed at the end of this manual.

Channel 1 is then used to control the flow rates in each channel. The CM will automatically set each other channel to the necessary flow rate based on the ratio set. As for the example, we set channel 1 equal to 41.4 SCCM, which sets channel 3 equal to 58.6 SCCM.

2.3 Totalizer Mode

The totalizer can be used to keep track of the amount of a certain amount of gas flowing through the mixing station. To change the totalizer settings from the starting screen, click DOWN, then select a channel, then TOTAL (DOWN). From here the totalizer mode can either be enabled or disabled using UP and DOWN respectively. After hitting NEXT (ESC), the starting threshold of the totalizer can be set. This sets a minimum percent flow rate needed to begin adding to the totalizer's total. This is useful because the command module generally measures a small amount of flow even when a channel is closed. Hitting NEXT (ESC) again allows you to set the totalizer stop value in the current engineering units. This is the value at which the totalizer stops counting and performs the set action. Hitting NEXT (ESC) one more time allows you to set the action to either NONE, BUZER, or VALVE. VALVE is particularly useful, as it will close the channel once the total is reached.

3 Other Command Module Parameters

3.1 Changing Density

If needed, the density of a gas being flowed through a channel can be set. From testing, it seems that setting the density only changes the calculations used to display the flow rate of a gas when using engineering units such as LBPH, LBPM, GrPH, GrPM. This is because the flow rate being measured is the volumetric flow, and the flow controllers have no way of actually measuring the amount of mass moving through them. Therefore, it is usually preferable to work with volumetric units, but if mass units are needed then the density can be changed by clicking DOWN, selecting a channel, then GENER. (ESC), then EXIT (ESC).

3.2 Changing Engineering Units

To change the engineering units being used for a channel, follow the same instructions to change the density but then click NEXT (ESC). From here UP and DOWN can be used to cycle through %FS, (percent flow rate based on a set value), SLPM (standard liters per minute), SLPH (standard liters per hour), SCCM (standard cubic centimeters per minute), SCCH (standard cubic centimeters per hour), SCFM (standard cubic feet per minute), SCFH (standard cubic feet per hour), SCMM (standard cubic meters per minute), SCMH (standard cubic meters per hour), LBPH (pounds per hour), LBPM (pounds per minute), GrPH (grams per hour), and GrPM (grams per minute). Generally, %FS, SLPM, and SCCM are the recommended units to use.

3.3 Setting the Percent Flow Rate Reference

This will only need to be changed when a new flow controller is connected to that channel of the CM. This value should be set to the maximum value that the flow controller is rated for and is the value from which all percent flow rate (%FS) values are based off of. To change this, follow the instructions to change the engineering units, then hit NEXT (ESC) one more time. The top of the screen should read CH# FS FLOW: #.### SLPM. Notice that the set engineering units do not change the fact that the percent flow rate is always based off of SLPM.

4 Troubleshooting

4.1 Calibrating for other gases

If a different gas is used in a channel other than the gas the MFC was calibrated with, the flow rate will not be correct. The MFCs are calibrated for a specific gas and use the thermal and chemical properties of the gas to set the valve height for the specified flow rate. If a new gas is needed, a calibration curve can be used to determine what the MFC needs to be set to in order to get the needed flow rate of the new gas. If one does not exist, a calibration curve can be made fairly easily.

Examples of calibration curves can be seen in section 5.1. The x axis shows the set flow rate in the MFC and the y axis shows the measured flow rate at the output of the channel. Using the trend line equation, the flow rate value to set in the MFC to get the necessary flow rate with the new gas can be calculated as $Q_{CalGas} = [Q_{NewGas} - b_{Fit}] / m_{Fit}$.

If a calibration curve needs to be made, this can be done by flowing the new gas through the channel needed and measuring its actual output for a variety of input values. Set the MFC to

flow various values from its low range to its high range. Using either a flow meter, or the bubble counter, measure the flow rate at each of the various set flow rates and plot the data in a scatter plot. The resulting trend line should be linear with a slope different from 1. Use this trend line to then calculate the necessary set MFC value to achieve the required output flow with the new gas.

4.2 Channel Flow Rate Fluctuations

It has been noticed that the channels are very sensitive to sudden fluctuations in the pressure of the gas lines. It is recommended to use higher pressure at the regulators and then slowly decrease the input pressure when gas is being flowed to the MFCs. When the MFCs receive a pressure wave, the solenoids will fluctuate and the flow rate value will start to fluctuate. It has been noticed that sometimes an MFC will enter a loop of oscillating over and under setting the flow rate. The best way to combat this is to allow the system to flow/get warm for at least 5-10 minutes between each step. For best results, I let the system warm up for 20 minutes at first. Then, I set each channel to its necessary value for the test and let all channels flow for 10 minutes before hooking up the hose to a detector. Then the system should be stable and accurate.

5 Calibration Curves

Here is the current collection of calibration curves that have been performed for the channels. The first three channels have been tested with both their calibration gas and a different gas. Channel four was not able to be tested with its calibration gas because we did not have Xe at the moment, instead channel 4 was tested with both Ar and CO₂ gas.

5.1 Flow Rate Calibration Curves

Each of these plots can be used to determine the flow rate the MFC needs to be set to to get a specific flow rate in this new gas. The trend line equation relates the set flow rate in the original gas, to the output flow rate with the new gas. Setting the trend line equation to the flow rate needed in the new gas and solving for x gives what value the MFC needs to be set to.

Flow Rate Calibration Curves for N2 and CO2 through Channel 1 MFC

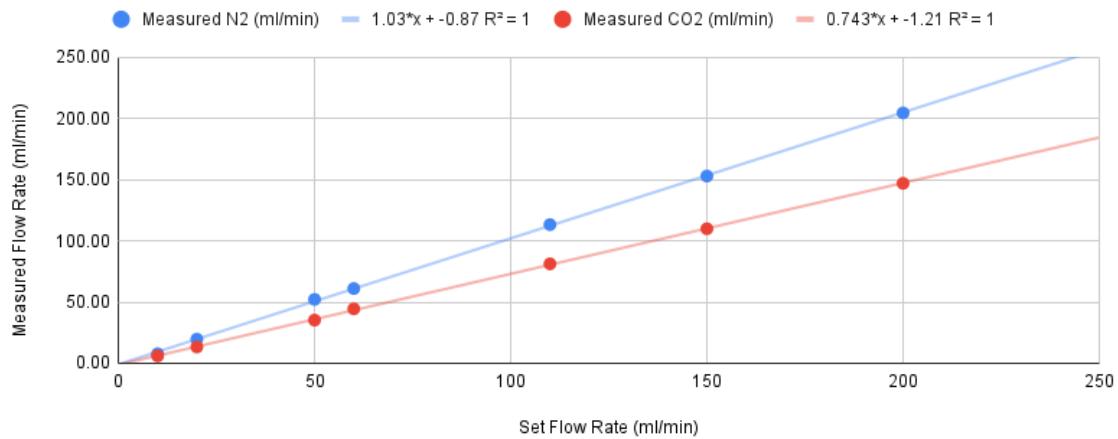


Figure 4: CO₂ and N₂ calibration curve for channel 1, which is factory calibrated for N₂.

Ch1 Calibration curve for N2 to Ar Gas

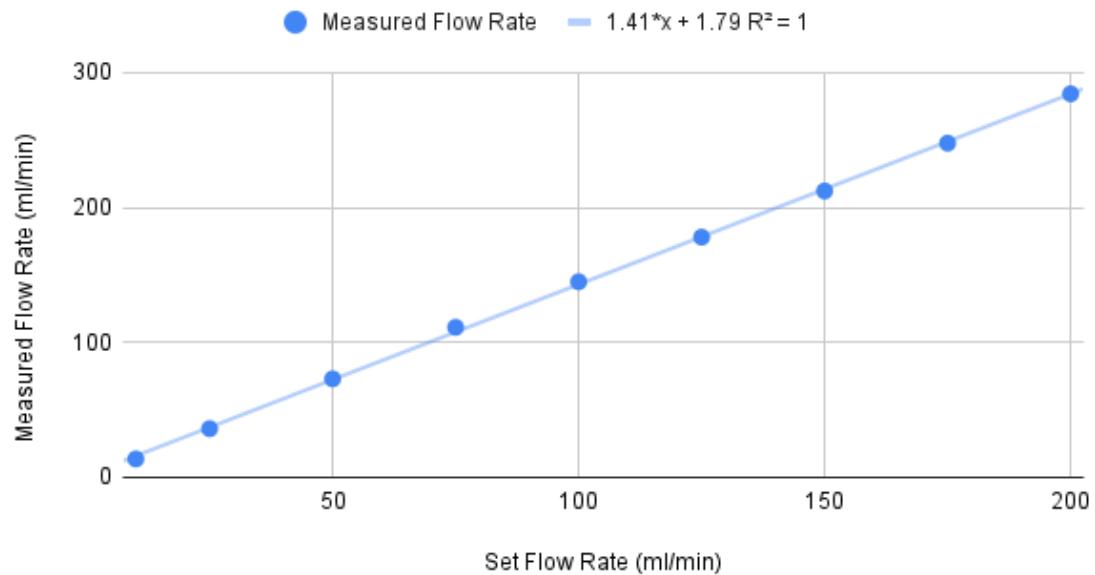


Figure 5: Argon calibration curve for channel 1, which is factory calibrated for N₂. Flow rates for both axes are given for Ar gas.

Flow Rate Calibration Curves for N2 and CO2 through Channel 2 MFC

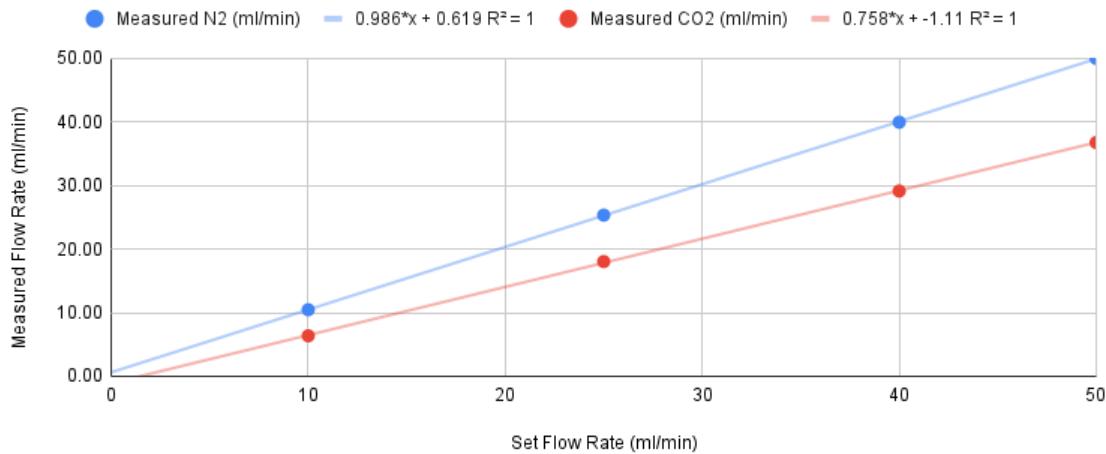


Figure 6: CO₂ and N₂ calibration curve for channel 2, which is factory calibrated for N₂.

Flow Rate Calibration Curves for Ar and N2 through Channel 3 MFC

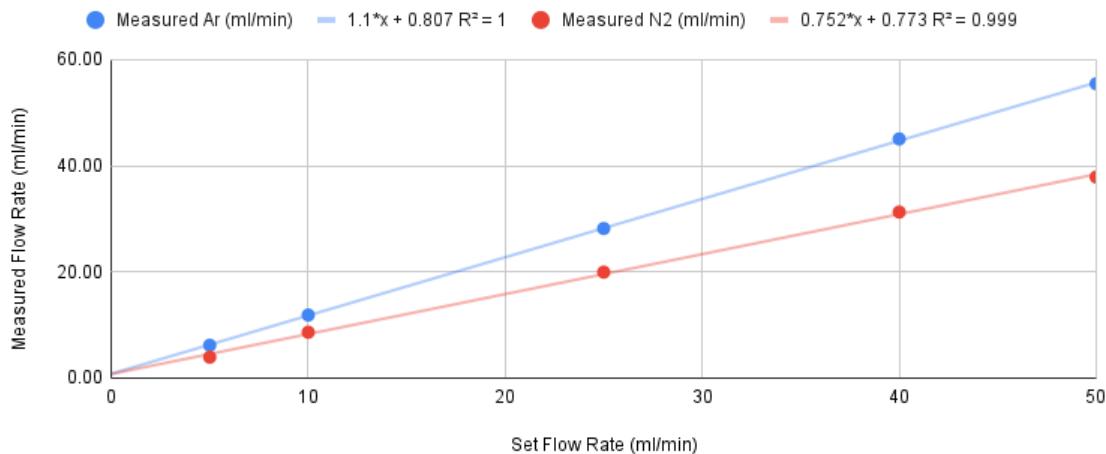


Figure 7: N₂ and Ar calibration curve for channel 3, which is factory calibrated for Ar.

Ch3 Calibration Curve for Ar to CO₂

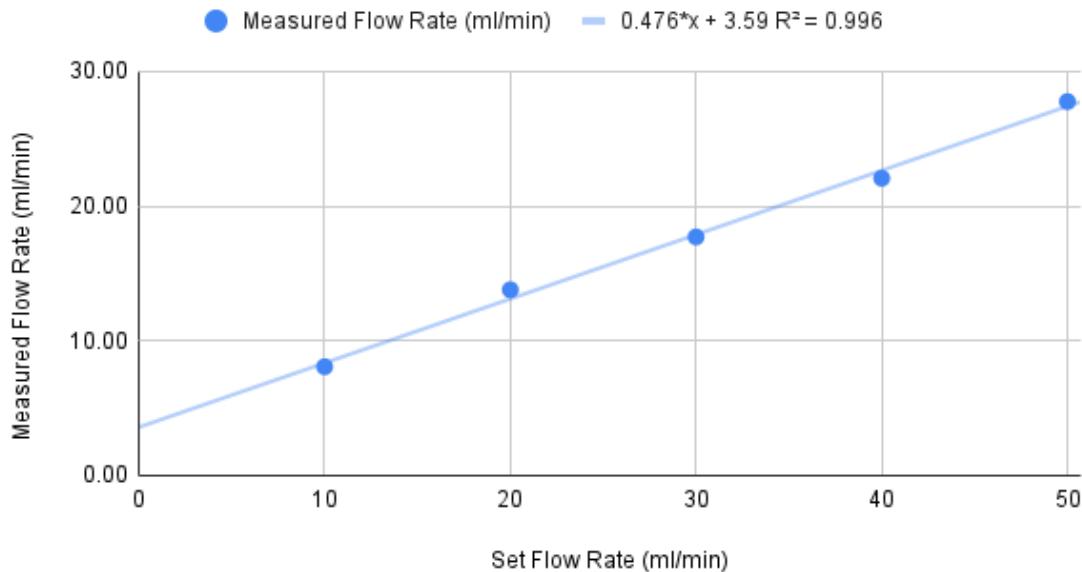


Figure 8: CO₂ calibration curve for channel 3, which is factory calibrated for Ar. Flow rates for both axes are given for CO₂ gas.

5.1.1 Creating Flow Rate Curves

There are multiple methods through which one could create a flow rate curve for a certain gas flowing through a specified channel. A simple but imprecise method would be to flow the gas into an inverted graduated cylinder filled with water and held underwater. Then one can measure the time it takes for the whole volume of the graduated cylinder to be displaced for various set flow rates.

If available, a similar but more accurate method would be to flow the gas into a Bubble-O-Meter and record the time it takes for the bubble to travel through a certain volume. The speed of the bubble depends on the radius of the section it is being pushed through, so three different sections with volumes of 1, 10, and 100 mL are available. The time it takes for the bubble to be displaced a certain volume is proportional to the gas flow rate, and the Bubble-O-Meter's manual provides the equation through which the conversion can be made for any given temperature or pressure. An example of gas displacing the bubbles in a Bubble-O-Meter is shown in Figure 9.

Whatever method is used to measure flow rates, the flow rate calibration curve can then be created by plotting the measured flow rate against the set flow rate. Ideally, a linear fit should be able to be applied to this curve.



Figure 9: Example of flow rate measurements being performed with a Bubble-O-Meter. The section with a volume of 10 mL can be seen between the two red rings. Currently in the picture, two bubble membranes can be seen flowing upward through this section. Above the highest red ring the 100 mL section starts, and many bubbles can be seen in this section.

5.2 Ratio Calibration Curves

These calibration curves are to bias the input ratio based on the machines previous ability to create these ratios. If one of the MFCs tend to increase its flow rate, then the ratio would need to be set in such a way to counteract the machines tendency to change the final ratio. These plots can be used in a similar way as the previous section, based on what ratio you want to set, use the trend line equation to find the new ratio to use for more accurate results.

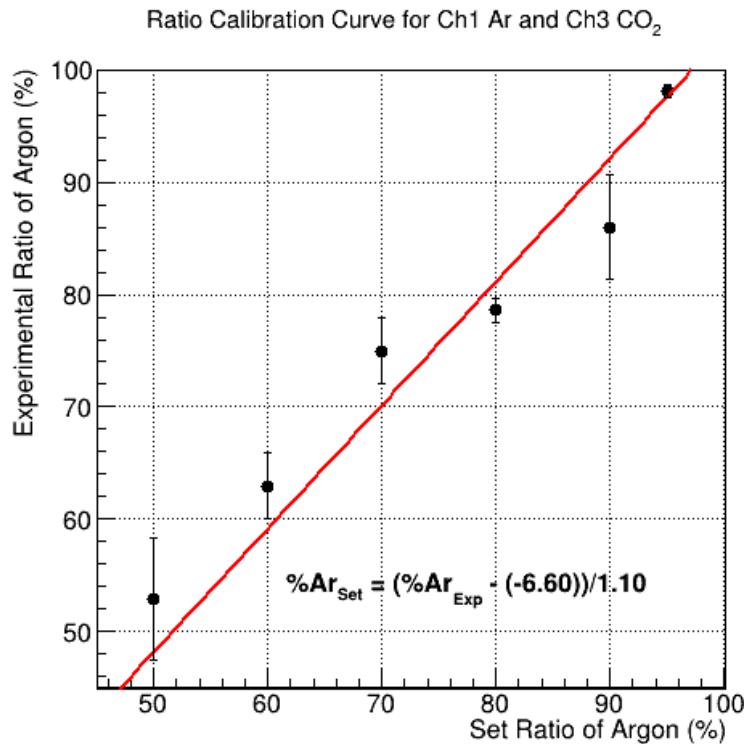


Figure 10: Ratio Calibration curve for Argon:CO₂ in Ch1:Ch3.

5.2.1 Creating Ratio Calibration Curves

One method from which to calculate the ratio of two gasses is to simply weigh the gas mixture and compare its mass to the mass of the pure gasses. First you must find the weight of the two pure gasses in the same volume as the container that will be used and at atmospheric pressure and room temperature. This can be done either through calculation or by experimentally weighing the pure gasses. Then you must weigh the mixture with the desired ratio to be tested. When filling a container with gas, use multiple volume changes (around a minimum of 10) worth of gas to ensure it is as pure as possible. Then place the container on a highly sensitive scale and record the mass every minute as it reaches pressure equilibrium with the atmosphere.

To do this, you must first create an apparatus which gas can flow in to while slowly pushing out old gas. For example, a corked flask with two syringe needles, one for the entering gas and one

for the exiting gas. The apparatus and scale used in our experiments can be seen in Figure 11. Alternatively, if one has access to a sealed container with a pressure gauge that gas can be flowed in to and that can be emptied with a vacuum pump, this may be better suited to the job.

We used a scale sensitive to 0.1 mg when the mass of the flask was included or 0.01 mg without the flask mass, and recorded the mass over time as the over pressured gas flowed out and reached pressure equilibrium with the atmosphere. We also recorded the temperature and external pressure for each measurement. To determine the mass, an exponential fit was applied to the mass over time and then the y-offset was measured. If the scale was tared before putting the flask on it, then the mass of the flask itself needs to be subtracted to get the mass of just the gas. An example trial can be seen in Figure 12. In this example, it can be seen that the exponential trend stops near 40 minutes and the rate of mass loss begins to increase. If our assumptions are correct, this signifies the point at which the flask has reached equilibrium and air has begun to diffuse into the flask. Therefore, only data up to this point is fitted.

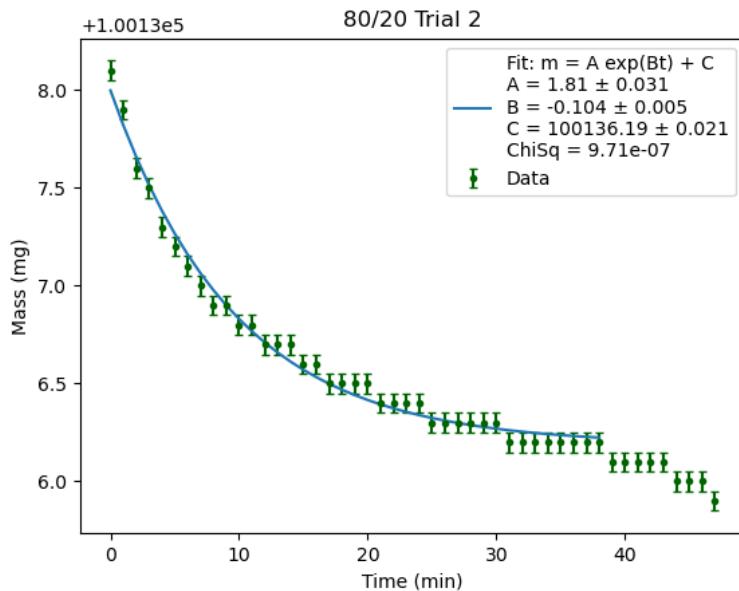


Figure 12: Example of applying an exponential fit to mass measurements of a mixture set to contain 80% Argon and 20% CO₂ as it reached equilibrium over time. Mass values are given as $+1.0013 \times 10^5$ mg. In this case, the scale was tared before setting the flask on it.

If known gas A and gas B are being mixed, the mole fraction (which is equal to the volume ratio according to Avogadro's Law) of gas A can be given by

$$X_A = \frac{m_{mix} - m_B}{m_A - m_B} \quad (1)$$

where m_{mix} is the measured mass, and m_A and m_B are the masses of gas A and gas B at the same temperature and pressure within the same volume as the measured mixed gas. This is of

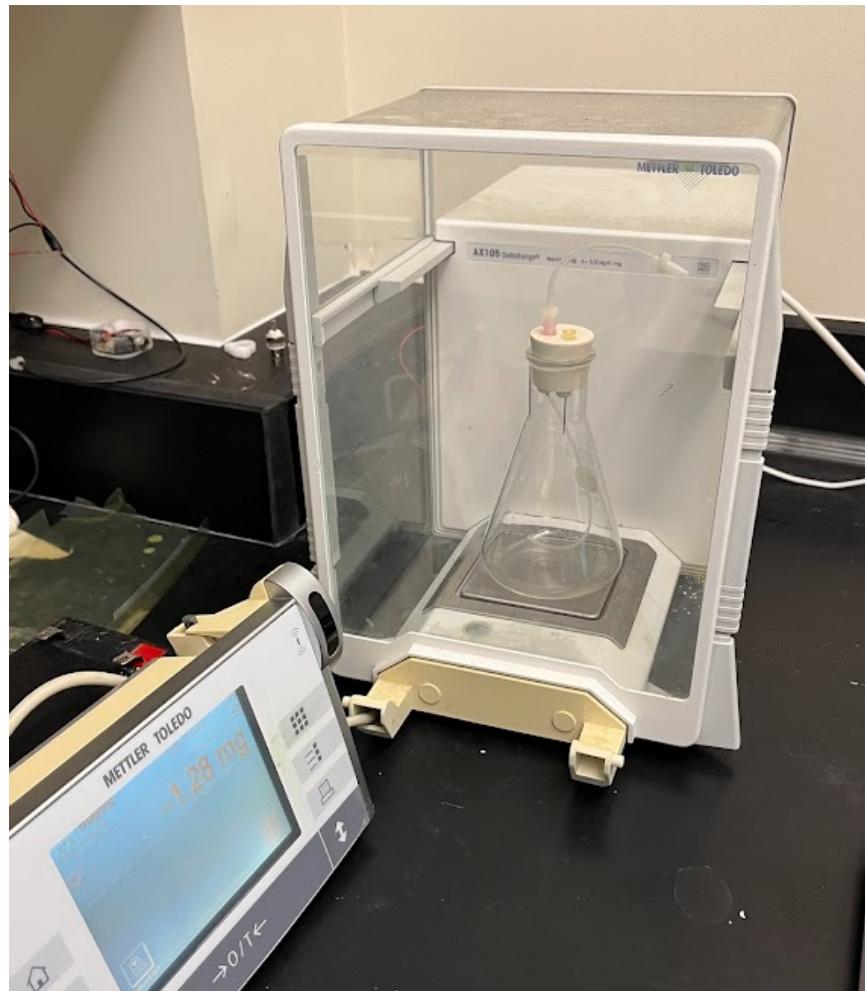


Figure 11: Gas weighing apparatus placed inside a highly sensitive mass scale.

course also assuming ideal gas properties. The mole fraction of gas B can then simply be found by $X_B = 1 - X_A$. After performing these calculations on a measured mixture, the measured ratios can be compared to the ratio that was set on the mixing station to determine how accurate the station was. After taking multiple such measurements, a ratio calibration curve can be plotted.

However, there are some downsides of this method, first being that it usually takes a long time (typically an hour) for the over pressured flask to reach pressure equilibrium. One way to get around this problem is by also measuring the mass of the pure gasses A and B in the flask, but this can only work if the temperature and pressure can be kept constant over all three total measurements (at least 3 hours of time) which is usually not feasible. Nevertheless, some tentative results obtained from this method can be found in Table 2 and an incomplete calibration curve for ArCO₂ mixtures using Channels 1 and 2 respectively is given by Figure 13.

| Channels | Input Ratio | Avg Measured Ratio | σ_{mean} |
|----------|-------------|--------------------|-----------------|
| 1 and 2 | 50% | 48.9% | 1.5% |
| 1 and 2 | 70% | 71.2% | 1.5% |
| 1 and 2 | 80% | 82.8% | 0.4% |
| 1 and 3 | 70% | 71.2% | 0.5% |
| 1 and 3 | 20% | 20.5% | 1.3% |

Table 2: Ratios are given as the percent Ar in an ArCO₂ mixture.

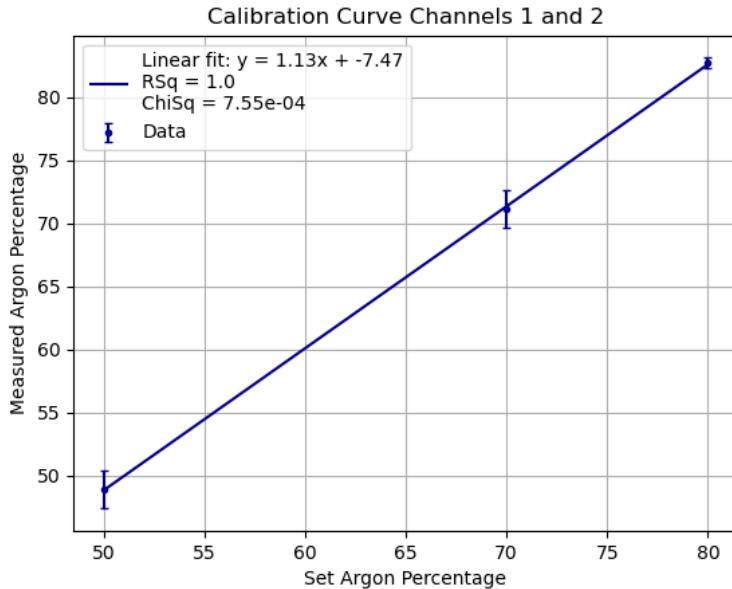


Figure 13: Incomplete calibration curve of channels 1 and 2 for mixtures of ArCO₂.